IS 3708-5 (2005): Methods of Test for Natural Rubber Latex, Part 5: Determination of KOH-number [PCD 13: Rubber and Rubber Products]

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IS 3708 (Part 5) : 2005
ISO 127 : 1995

भारतीय मानक

प्राकृतिक रबड़ लेटैक्स की परीक्षण पद्धतियाँ

भाग 5 के.ओ.एच. संख्या ज्ञात करना

(दूसरा पुनरीक्षण)

Indian Standard

METHODS OF TEST FOR NATURAL RUBBER LATEX

PART 5 DETERMINATION OF KOH NUMBER

(Second Revision)

ICS 83.040.10

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 2005 **Price Group 3**

NATIONAL FOREWORD

This Indian Standard (Part 5) (Second Revision) which is identical with ISO 127 : 1995 'Rubber, natural latex concentrate — Determination of KOH number' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendations of the Rubber and Rubber Products Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The text of ISO Standard has been proposed to be approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

The concerned Technical Committee has reviewd the provisions of the following International Standards referred in this adopted standard and has decided that they are acceptable for use in conjunction with this standard:

International Standard	*Title*
ISO 123 : 2001	Rubber latex — Sampling
ISO 124 : 1997	Latex, rubber — Determination of total solids content
ISO 125 : 1990	Natural rubber latex concentrate — Determination of alkalinity
ISO 976 : 1996	Rubber and plastics — Polymer dispersion and rubber latices — Determination of *p*H
ISO 1802 : 1992	Natural rubber latex concentrate — Determination of boric acid content

For tropical countries like India, the standard temperature and the relative density shall be taken as 27 ± 2°C and 65 ± 5 percent relatively.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

METHODS OF TEST FOR NATURAL RUBBER LATEX

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(Second Revision)

WARNING — Persons using this standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies a method for the determination of the KOH number of natural rubber latex concentrate which is preserved wholly or in part with ammonia. The method is applicable to latices containing boric acid. The method is not applicable to latices preserved with potassium hydroxide. It is not necessarily suitable for latices from natural sources other than *Hevea brasiliensis,* or for latices of synthetic rubber, compounded latex, vulcanized latex or artificial dispersions of rubber.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 123:1985, *Rubber latex — Sampling.*

ISO 124:1992, *Rubber latices — Determination of total solids content.*

ISO 125:1990, *Natural rubber latex concentrate — Determination of alkalinity.*

ISO 976:—[1], *Rubber latices — Determination of pH.*

ISO 1802:1985, *Natural rubber latex concentrate — Determination of boric acid content.*

3 Definition

For the purposes of this International Standard, the following definition applies.

3.1 KOH number (of latex): The number of grams of potassium hydroxide equivalent to the acid radicals combined with ammonia in latex concentrate containing 100 g of total solids.

(Definition taken from ISO 1382:1982, *Rubber — Vocabulary*/Add.2:1982.)

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water free of dissolved carbon dioxide, or water of equivalent purity.

1) To be published.

4.1 Potassium hydroxide, standard volumetric solution, $c(KOH) = 0,1$ mol/dm^3, carbonate-free.

4.2 Potassium hydroxide, standard volumetric solution, $c(KOH) = 0,5$ mol/dm^3, carbonate-free.

4.3 Formaldehyde, 45 g to 50 g in 1 dm^3 of solution [$c(HCHO) = 1,5$ mol/dm^3 to 1,67 mol/dm^3], acid-free, prepared by diluting concentrated formaldehyde with water and neutralizing with 0,1 mol/dm^3 potassium hydroxide solution (4.1), using as indicator the faint pink colour of phenolphthalein.

Determine the concentration of the formaldehyde solution as described in annex A.

5 Apparatus

Standard laboratory glassware, plus the following:

5.1 pH-meter, conforming to ISO 976 but capable of being read to 0,01 units.

5.2 Glass electrode, of a type suitable for use in solutions of pH up to 12,0.

5.3 Mechanical stirrer, with earthed motor and glass paddle, or **magnetic stirrer**.

NOTE 1 An automatic titrator may be used provided it has been checked as giving the same result as the standard method.

6 Sampling

Carry out the sampling in accordance with one of the methods specified in ISO 123.

7 Procedure

Calibrate the pH-meter by the method specified in ISO 976. If the total solids (w_{TS}) and alkalinity (A) of the latex are not known, determine them in accordance with ISO 124 and ISO 125 respectively. If the latex contains boric acid and the content is not known, determine it in accordance with ISO 1802.

Carry out the determination in duplicate.

Weigh, to the nearest 0,1 g, into a 400 cm^3 beaker a test portion (mass m) of the test sample containing approximately 50 g of total solids. If necessary, adjust the alkalinity to (0,5 ± 0,1) % ammonia calculated with respect to the water phase by adding, while stirring, the required quantity of formaldehyde solution (4.3).

Calculate the volume of formaldehyde solution to be added from the formula

$$\frac{m(100 - w_{TS})(A - 0,5)}{1134\,c(\text{HCHO})}$$

where $c(\text{HCHO})$ is the actual concentration, expressed in moles per cubic decimetre, of the formaldehyde solution (4.3).

Dilute the latex with water to about 30 % total solids.

Insert the electrodes of the pH-meter (5.1) into the diluted latex concentrate and record the pH.

If the initial pH is less than 10,3, slowly add 5 cm^3 of 0,5 mol/dm^3 potassium hydroxide solution (4.2) while stirring slowly with the glass paddle or magnetic stirrer (5.3). Record the resultant equilibrium pH reading. With continued stirring, add 0,5 mol/dm^3 potassium hydroxide solution (4.2) in 1 cm^3 increments at regular (e.g. 15 s) intervals, recording the resultant equilibrium pH after each addition. Continue until the end-point has been passed.

If the initial pH is 10,3 or higher, omit the initial addition of 5 cm^3 at one time and proceed directly to "add 0,5 mol/dm^3 potassium hydroxide solution (4.2) in 1 cm^3 increments" as described above.

The end-point of the titration is the point of inflexion of the titration curve of the pH-value against the volume, in cubic centimetres, of potassium hydroxide solution. At this point, the slope of the curve, i.e. the first differential, reaches a maximum and the second differential changes from a positive to a negative value. The end-point shall be calculated from the second differential on the assumption that the change from a positive to a negative value bears a linear relation to the addition of potassium hydroxide during the 1 cm^3 interval involved.

An example of a typical titration and the calculation of the end-point is given in annex B.

The results of duplicate determinations shall agree to within 5 % (m/m).

8 Expression of results

Calculate the KOH number, expressed as a percentage by mass, of the latex concentrate from the formula

$$\frac{561\, c \cdot V}{w_{TS}\, m}$$

where

c is the actual concentration, expressed in moles of KOH per cubic decimetre, of the potassium hydroxide solution (4.2);

V is the volume, in cubic centimetres, of the nominally 0,5 mol/dm^3 potassium hydroxide solution (4.2) required to reach the end-point;

w_{TS} is the total solids content, expressed as a percentage by mass, of the latex concentrate;

m is the mass, in grams, of the test portion.

If the latex concentrate contains boric acid, subtract the KOH number equivalent to the boric acid from the KOH number obtained above. Calculate the KOH number equivalent to the boric acid present from the formula

$$91 \times \frac{w_{BA}}{w_{TS}}$$

where w_{BA} is the boric acid content, expressed as a percentage by mass.

9 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for complete identification of the sample;

c) all details necessary for complete identification of the pH-meter used;

d) the result obtained;

e) the correction applied for boric acid, if present;

f) details of any operation not included in this International Standard or regarded as optional;

g) the date of the test.

Annex A
(informative)

Determination of formaldehyde

The method given in previous editions of this International Standard appears not to have been widely used due to the fact that standard solutions of ammonia are considered to be unsatisfactory. Having regard to the consistent quality of analytical-grade concentrated formaldehyde solution, the majority of users prepare a standard solution of formaldehyde directly.

Where it is necessary to determine the concentration of the diluted formaldehyde, a variety of methods exist and users are referred to the *Encyclopaedia of Industrial Chemical Analysis*, Vol. 13, published by Interscience (1971). The method given below is for information only.

A.1 Reagents

A.1.1 Sodium sulfite, anhydrous, analytical grade.

A.1.2 Sulfuric acid, standard volumetric solution, $c(H_2SO_4) = 0{,}25$ mol/dm^3.

A.1.3 Thymolphthalein, indicator solution.

Dissolve 80 mg of thymolphthalein in 100 cm^3 of ethyl alcohol and dilute with 100 cm^3 of distilled water.

A.2 Procedure

Prepare a solution of 125 g of anhydrous sodium sulfite (A.1.1) in 500 cm^3 of water and dilute to 1 dm^3. Transfer 100 cm^3 of the solution to a 500 cm^3 conical flask. Accurately weigh into the flask 6,0 g to 8,0 g of the nominally 50 g/dm^3 formaldehyde solution (4.3) [2] and swirl to mix thoroughly. Allow to stand for 5 min, then titrate with 0,25 mol/dm^3 sulfuric acid (A.1.2) to the first colourless end-point using thymolphthalein (A.1.3) as indicator. Run a blank determination with the soldium sulfite solution.

A.3 Expression of results

Calculate the formaldehyde content, expressed as a percentage by mass, of the formaldehyde solution from the formula

$$\frac{30{,}03(V_1 - V_2) \times 2c(H_2SO_4)}{10m_1}$$

where

V_1 is the volume, in cubic centimetres, of sulfuric acid (A.1.2) used for the titration of the test portion of formaldehyde solution;

V_2 is the volume, in cubic centimetres, of sulfuric acid (A.1.2) required for the blank;

$c(H_2SO_4)$ is the actual concentration, in moles of H_2SO_4 per cubic decimetre, of the sulfuric acid;

m_1 is the mass, in grams, of the test portion of formaldehyde solution.

2) When analysing concentrated formaldehyde solution, 1,8 g to 2,0 g of solution is a more convenient amount to take.

Annex B
(informative)

Example of a typical titration and calculation of end-point

Table B.1 — Example of a typical titration to show variation in pH

Volume of KOH solution added cm^3	**pH reading**	**First difference** $\Delta pH/cm^3$	**Second difference** $\Delta^2 pH/cm^3$
Initial	10,09		
5	10,46		
		0,09	
6	10,55		0,01
		0,10	
7	10,65		0,01
		0,11	
8	10,76		0,03
		0,14	
9	10,90		0,04
		0,18	
10	11,08		0,06
		0,24	
11	11,32		0,07
		0,31	
12	11,63		−0,01
		0,30	
13	11,93		−0,09
		0,21	
14	12,14		

In this example, the first difference reaches a maximum of 0,31 between 11 cm^3 and 12 cm^3 of potassium hydroxide solution. The precise point of inflexion is calculated from the ratio of the adjacent second-difference values, i.e. 0,07/(0,07 + 0,01) = 0,875 of the difference between 11 cm^3 and 12 cm^3, i.e. 11,875 cm^3.

Figure B.1 presents this data graphically to show the point of inflexion.

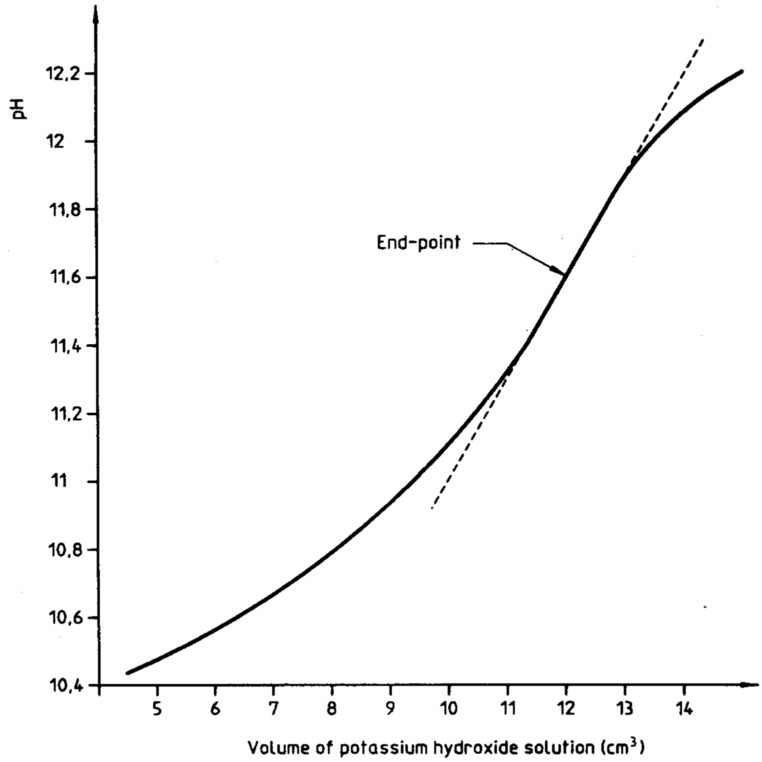

Figure B.1 — Illustrative curve showing change of pH during titration

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

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